

### **Section III: Identification of Heroin**

#### *I. Introduction:*

Heroin samples are screened and analyzed by GC/FID and subsequently confirmed by GC/MS.

#### *II. Reagents:*

- A.) 9:1 Methylene Chloride/Isopropanol, Ethanol, or Methanol.
- B.) Methanol (solvent rinse for GC)
- C.) 0.1 N HCL: add 8.3 mL concentrated HCL to sufficient water to make 1 L. (quantitation).
- D.) Benzopinacolone and 1.0 mg/mL heroin (quantitation standard).
- E.) 10% K<sub>2</sub>HPO<sub>4</sub>: dissolve 10g K<sub>2</sub>HPO<sub>4</sub> in sufficient water to make 100 mL. (quantitation).
- F.) 1.0 mg/mL Benzopinacolone in Methylene chloride (quantitation internal standard).
- G.) Anhydrous Sodium Sulfate (Na<sub>2</sub>SO<sub>4</sub>).

#### *III. Equipment:*

- A.) Analytical balance
- B.) Weigh paper
- C.) Pipettes
- D.) 25 mL volumetric flask
- E.) Stoppered test tube
- F.) 2 mL autosampler vials with Teflon caps
- G.) GC/FID: HP 6890 or 7890A
- H.) GC/MS: HP 5890/5972 or HP 6890/5973 series.

#### *IV. Procedure:*

- A.) Chromatography by GC/FID and GC/MS.
  - 1. Obtain gross weight of sample evidence bag.
  - 2. Remove one sample packet from the evidence bag and weigh.
  - 3. Record individual packet gross weight in logbook.
  - 4. Remove powder from packet onto a weigh paper.
  - 5. Reweigh the empty packet and record the weight.
  - 6. Add about 5 mg of sample to a labeled 2 mL autosampler vial.
  - 7. Add powder back to packet and reseal.

8. Return packet(s) to original evidence back, reseal, and then put evidence bag in a new plastic bag and seal.
9. Subtract empty packet weight from total weight to obtain the powders net weight.
10. For up to 100 packets, analyze 10% of the total. Report the average of the individual the net weights.
11. For more than 100 packets, analyze the square root of the total. Report the average of all the individual net weights.
12. Add 1-2 mL of Ethanol, Methanol or 9:1 Methylene Chloride/Isopropanol to the autosampler vial(s) containing the sample and cap.
13. Place vial(s) on the GC/FID autosampler and run with the following sequence: Standard, Blank, Samples.
14. GC/FID conditions are as follows:

Method: EXP.M

Oven:

Initial Temp: 245°C  
Initial Time: 0.00 min.  
Rate: 10°/min.  
Final Temp: 290°C  
Run Time: 10 min.  
Max. Temp: 325°C  
Equilibration Time: 0.5 min.

Inlet:

Mode: split (35:1)  
Initial Temp: 250°C  
Pressure: 24.99 psi  
Gas Type: Helium

Column:

Capillary: HP-1 30m x 320um  
Initial Flow: 3.3 mL/min.

Detector:

Temp: 300°C  
Hydrogen Flow: 30.0 mL/min.  
Air Flow: 400 mL/min.  
Makeup Gas: Helium

15. Obtain chromatographs. If heroin is present, the instrument will detect a peak with a retention time around 5.7 minutes and will generate a report with accompanying chromatograph.
16. Check concentration to determine if a dilution is needed or if the injection volume needs to be increased for subsequent GC/MS run. Also observe any erroneous data that indicates the sample may have to be reinjected.

17. Place same sequence on the GC/MS autosampler and run.
18. GC/MS conditions are as follows:  
Method: EXP.M  
Oven:  
    Initial Temp: 230°C  
    Initial Time: 0.00 min.  
    Max. Temp: 325°C  
    Equilibration Time: 0.50 min.  
    Rate: 10°/min.  
    Final Temp: 280°C  
    Run Time: 10 min.  
Inlet:  
    Mode: split (50:1)  
    Initial Temp: 250°C  
    Pressure: 31.65 psi  
    Gas Type: Helium  
Column:  
    Capillary: HP-1MS 25m x 200um x 0.33um  
    Max. Temp: 300°C  
    Initial Flow: 1.0 mL/min.
19. If heroin is present in sample, the instrument will detect a total ion peak at approximate retention time of 6.4 minutes and will generate a report along with accompanying chromatograph and spectra. The spectra will contain the identity of the peak and its ion abundance.

B.) Quantitation Procedure:

1. Extract sample by weighing out 100 mg of sample in a 25 mL volumetric flask and bring to volume with 0.1 N HCL.
2. Record exact weight and calculate sample amount by dividing the weight by the final volume.
3. The heroin standard is prepared and should contain about 1.0 mg/mL of Benzopinacolone and 1.0 mg/mL of heroin.
4. The internal standard is 1.0 mg/mL Benzopinacolone in Methylene Chloride.
5. In a stoppered test tube, labeled with sample number, add 2 mL of sample (prepared in step#1), 2 mL of 0.1 N HCL, and 1 mL of K<sub>2</sub>HPO<sub>4</sub>. Two layers will form in tube.
6. In a labeled autosampler vial, add enough sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>) to cover the bottom of the vial.

7. From the sample test tube, pipette the bottom layer into the appropriate vial and cap.
8. Run the quantitation on the GC, with the following sequence: Heroin Standard (inj.#1), Heroin Standard (inj.#2, Calibration), Heroin Standard (inj.#3), Blank, Sample(s), Heroin Standard (inj#4).
9. Check the standard to make sure recovery is at 100%. If not, rerun the standard (possibly at a higher injection amount).

*V. Results:*

- A.) Record results of the GC/MS in logbook. Then transfer the results to appropriate evidence cards that came with the actual samples. Be sure to include date of analysis, results, the number of tests performed per sample, and initials.
- B.) All reports generated from the instruments should be filed so that they may be accessed at a later date, if necessary.